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IS 12004 (1987): Isoproturon Technical [FAD 1: Pesticides and Pesticides Residue Analysis]



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“Knowledge is such a treasure which cannot be stolen”

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REAFFIRMED

-- ~~1987~~ 2002

IS : 12004 - 1987

Indian Standard
**SPECIFICATION FOR
ISOPROTURON TECHNICAL**

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**BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002**

Indian Standard

SPECIFICATION FOR ISOPROTURON TECHNICAL

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**AMENDMENT NO. 2 MAY 2002
TO
IS 12004 : 1987 SPECIFICATION FOR ISOPROTURON
TECHNICAL**

(Page 4, clause 2.2) — Delete.

(FAD I)

Reprography Unit, BIS, New Delhi, India

Indian Standard

SPECIFICATION FOR ISOPROTURON TECHNICAL

0. FOREWORD

0.1 This Indian Standard was adopted by the Bureau of Indian Standards on 28 May 1987, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council and the Chemical Division Council.

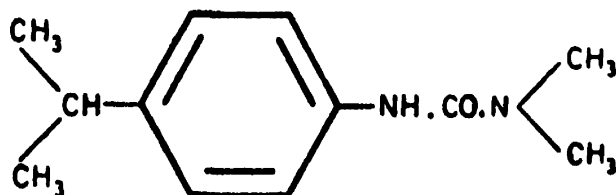
0.2 Isoproturon, technical is used for making formulations meant for weed control in agricultural crops.

0.3 Isoproturon is the accepted common name by the International Organisation for Standardization (ISO) for *N,N*-dimethyl-*N*-4-isopropyl-phenyl urea. The empirical and structural formulae, and molecular mass are as given below:

Empirical Formula
 $C_{12}H_{18}N_2O$

Structural Formula

Molecular Mass
206.29



0.4 In the preparation of this standard, due consideration has been given to the provisions of the *Insecticides Act*, 1968, and Rules framed thereunder. However, this standard is subject to the restrictions imposed under the Act and Rules, wherever applicable.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Rules for rounding off numerical values (revised).

1. SCOPE

1.1 This standard prescribes the requirements, and the methods of sampling and test for isoproturon, technical.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of white to greyish white or yellowish crystalline powder free from extraneous impurities or hard lumps.

2.2 Identity Test — Extract about 1 g of the sample in about 50 ml methanol. Centrifuge for about 5 minutes. Decant the clear supernatant liquid into a beaker. Add 250 to 300 ml of water to precipitate the isoproturon. Filter, wash thoroughly with water and dry at 100°C. Melting point of dry residue shall be between 154-156°C and shall not be depressed by the addition of pure isoproturon. Further IR spectrum of the residue taken in potassium bromide should be superimposable on the IR spectrum of the reference sample of isoproturon between 2.5 and 15 μ .

NOTE — Identity test need not be carried out if isoproturon content is determined by UV spectrophotometric method (*see* A-1) or HPLC method (*see* A-2).

2.3 The material shall also comply with the requirements specified in Table 1.

TABLE 1 REQUIREMENTS FOR ISOPROTURON TECHNICAL

| Sl No. | CHARACTERISTIC | REQUIREMENT | METHOD OF TEST, REF TO | |
|--------|--|-------------|---------------------------------|----------------------------------|
| | | | Appendix of this Standard | Cl No. of IS : 6940- 1982* |
| (1) | (2) | (3) | (4) | (5) |
| i) | Isoproturon content, percent by mass, <i>Min</i> | 95.0 | A | — |
| ii) | Water content, percent by mass, <i>Max</i> | 0.5 | — | 4 |
| iii) | Melting point, °C | 152-158 | — | 6 |
| iv) | Acidity (as H ₂ SO ₄), percent by mass, <i>Max</i> | 0.1 | — | 11.3 |
| | <i>or</i> | | | |
| v) | Alkalinity (as NaOH), percent by mass, <i>Max</i> | 0.4 | — | 11.3 |

*Methods of test for pesticides and their formulations (*first revision*).

3. PACKING AND MARKING

3.1 Packing — The material shall be packed according to the requirements given in IS : 8190 (Part 1)-1980*.

3.2 Marking — The container shall bear legibly and indelibly the following information and any other information as is necessary under the *Insecticides Act* and Rules:

- a) Name of the material;
- b) Name of the manufacturer or trade-mark;
- c) Batch number;
- d) Date of manufacture;
- e) Isoproturon content, percent (*m/m*);
- f) Net mass of the contents; and
- g) The cautionary notice as worded in the *Insecticides Act* and Rules.

3.2.1 The container may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

4. SAMPLING

4.1 Representative samples of material shall be drawn as prescribed in IS : 10946-1984†.

5. TESTS

5.1 Tests shall be carried out as referred to in col 4 and 5 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977‡) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

*Requirements for packing of pesticides: Part 1 Solid pesticides (*first revision*).

†Methods of sampling for technical grade pesticides.

‡Specification for water for general laboratory use (*second revision*).

APPENDIX A

[Table 1, Item (i)]

DETERMINATION OF ISOPROTURON CONTENTS

A-0. GENERAL

A-0.1 For the determination of isoproturon content, three methods namely. UV spectrophotometric method (*see A-1*), HPLC method (*see A-2*) and basic hydrolysis method (*see A-3*) have been specified. UV spectrophotometric method and HPLC method shall be the referee method in case of dispute.

A-1. UV SPECTROPHOTOMETRIC METHOD

A-1.1 Principle — The absorbance of a methanolic solution of technical material is measured against the solvent at 242 nm. The isoproturon content of the sample is then computed making use of the absorbance value of a solution of standard isoproturon.

A-1.2 Apparatus

A-1.2.1 Ultraviolet Spectrophotometer

A-1.2.2 Quartz Cells — Matched pair with path length equal to 1.000 cm.

A-1.2.3 Volumetric Flasks — 250-ml, 200-ml and 100-ml capacity.

A-1.2.4 Pipettes — 10-ml and 5-ml capacity (graduated).

A-1.3 Reagents

A-1.3.1 Methanol — Spectroscopic grade.

NOTE — The absorbance (1.000 cm cell) of methanol should not, in any case, exceed 0.150 at 240 nm. The spectral absorbance curve should be smooth throughout the 210-300 nm range and should not show any extraneous impurity peaks.

A-1.4 Procedure

A-1.4.1 Weigh out accurately about 150 mg of the standard isoproturon material into a dry 250-ml flask, dissolve and dilute to volume with methanol. Pipette out 10 ml of this solution into a 100-ml flask and dilute to volume with methanol. Pipette out 10 ml of this solution into a third flask of 100-ml capacity and dilute to volume. This final solution is taken for absorbance measurement. Weigh out accurately about 150 mg of the isoproturon, technical sample to be analyzed, into a dry 250-ml flask and proceed exactly as suggested above for the standard preparation.

A-1.4.2 Spectrophotometric Determination — Measure the absorbance of the standard as well as sample solutions described under A-1.4.1 at 242 nm using the 1.000 cm cuvette and methanol as blank.

A-1.5 Calculations

$$\text{Isoproturon content, percent by mass} = \frac{m_1 \times A_2 \times P}{A_1 \times m_2}$$

where

m_1 = mass in mg of the standard taken,

A_2 = absorbance of the sample,

P = percentage purity of standard isoproturon,

A_1 = absorbance of the standard isoproturon, and

m_2 = mass in mg of the sample taken.

A-2. HPLC METHOD

A-2.1 Principle — A HPLC unit with a UV detector is used for this assay. Using a solution containing known amounts of the standard isoproturon sample and the internal standard, the response factor, RF , for isoproturon in the internal approach is arrived at. A solution containing a known mass of the isoproturon sample and internal standard is injected subsequently into the HPLC unit. The percentage of isoproturon in the sample is then computed by the standard relationship.

A-2.2 Apparatus

A-2.2.1 High performance liquid chromatograph equipped with a printer-plotter-cum-integrator and UV Detector. The suggestive HPLC operating conditions are given below. However, these operating conditions are likely to change with change in HPLC equipment employed and are allowed provided standardization is done:

| | |
|-------------------|--|
| Column | Silica, 10 μm 25 cm \times 4.6 mm (S.S) |
| Solvent system | a) Cyclohexane 90 percent (v/v) b) Isopropanol 10 percent (v/v) |
| Detector | UV (at 254 nm) |
| Solvent flow rate | 1.5 ml/min |
| Chart speed | 0.2 cm/min |
| Sample size | 10 μl |

A-2.2.2 Volumetric Flask — 50-ml and 100-ml capacity.

A-2.2.3 Pipettes (graduated) — 2-ml, 5-ml and 10-ml capacity.

A-2.3 Reagents

A-2.3.1 Internal Standard — acetanilide, A.R. or equivalent grade.

A-2.3.2 Cyclohexane — spectroscopic grade.

A-2.3.3 Isopropanol — spectroscopic grade.

A-2.3.4 Isoproturon — of known purity.

A-2.4 Preparation of the Standard and Sample Solutions

A-2.4.1 Weigh out accurately 0.25 g of acetanilide into a 100-ml volumetric flask and make up to volume using the cyclohexane-isopropanol mixture (90 : 10, v/v). This will give a solution containing 2.5 mg/ml of the internal standard.

A-2.4.2 Weigh out 0.5 g of standard isoproturon into a 100-ml volumetric flask and dissolve it in 25 ml isopropanol. Make up to volume using the cyclohexane. This will give a stock solution containing 5 mg/ml of the standard isoproturon. Pipette out 5 ml of this standard solution into a 50-ml volumetric flask. Then pipette out 5 ml of the internal standard solution into the same flask. Mix well. Make up to the mark using the solvent mixture. Call this, *solution A*. Weigh out 0.5 g of the isoproturon sample and proceed exactly as in the case of the standard sample. Call this, *solution B*.

A-2.5 Procedure

A-2.5.1 Introduce 10 µl of the *solution A* and *solution B* into the HPLC unit. From the integrator print out and note down the peak areas of the isoproturon and acetanilide peaks in both the cases. Adjust the attenuation in such a way that the isoproturon and acetanilide peaks are obtained within the scale in both the cases. (This attenuation may change from equipment to equipment). Compute the percentage of isoproturon content in the sample as indicated in A-2.6.

A-2.6 Calculations

$$\text{Isoproturon content, percent by mass} = \frac{m_1 \times A_2 \times P \times A_3}{A_1 \times m_2 \times A_4}$$

where

m_1 = mass of standard isoproturon in *solution A*;

A_2 = area of isoproturon peak in *solution B*;

P = percentage purity of standard isoproturon;

A_3 = area of internal standard peak in *solution A*;

A_1 = area of standard isoproturon peak in *solution A*;

m_2 = mass of the isoproturon sample in *solution B*;

A_4 = area of internal standard peak in *solution B*.

A-2.7 Precision — Data obtained by this method indicate a standard deviation of 0.5 for isoproturon at 96 percent level.

A-3. BASIC HYDROLYSIS METHOD

A-3.1 Principle — Isoproturon is hydrolysed using potassium hydroxide in aqueous diethylene glycol (1 : 1 by volume). Dimethylamine, the volatile product of hydrolysis, is distilled off and absorbed in standard hydrochloric acid. From the quantity of acid consumed by dimethylamine, the percentage of isoproturon present in the original sample is computed.

A-3.2 Apparatus

A-3.2.1 An all glass distillation assembly of a suitable type. A suggestive assembly is shown in Fig. 1.

A-3.3 Reagents

A-3.3.1 *Hydrochloric Acid Solution* — 0.2 N.

A-3.3.2 *Sodium Hydroxide Solution* — 0.2 N.

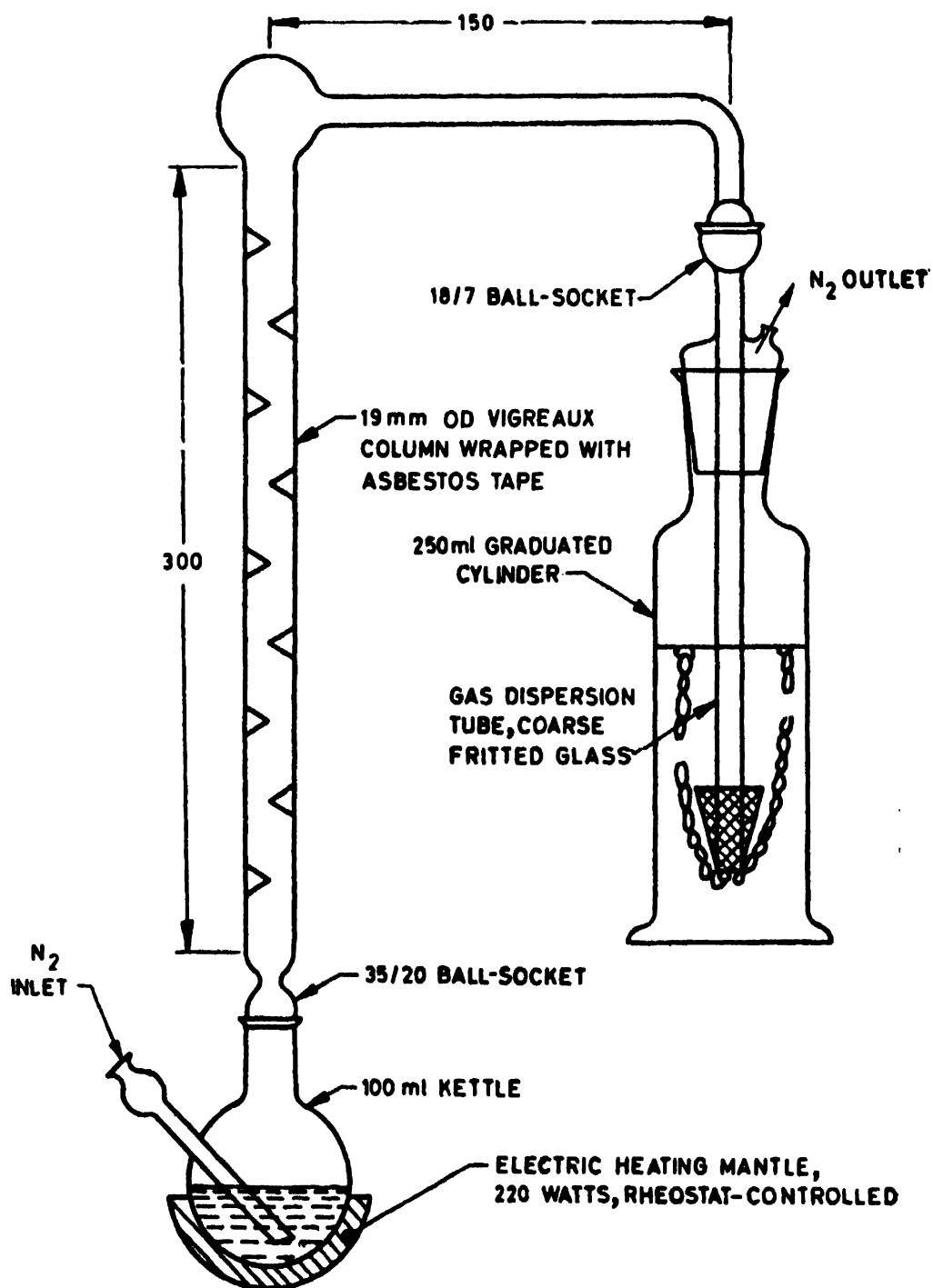
A-3.3.3 *Potassium Hydroxide Solution* — 35 percent aqueous.

A-3.3.4 *Diethylene Glycol*

A-3.3.5 *Silicon Defoamer*

A-3.4 Procedure

A-3.4.1 Accurately weigh 1.0 ± 0.1 g of the isoproturon, technical sample and transfer it quantitatively to a clean dry reaction flask of the hydrolysis-cum-distillation apparatus. Add 50-ml of diethylene glycol and one or two boiling chips. One ml of silicon defoamer may be added to check foaming. Attach the reaction flask to the other parts of the assembly as shown in Fig. 1. 50 ml of hydrochloric acid is taken in a beaker in which the delivery tube end dips as shown in Fig. 1. Add 50 ml of potassium hydroxide solution to the flask through a dropping funnel. Close the stop-cock of the dropping funnel as soon as the addition of alkali is complete. Heat the contents of the reaction flask to boiling. Continue the distillation up to about 2½ hours. The distillation can be stopped when the contents of the flask become orange brown in colour. The distillate will cease to be alkaline by that time. This can further be confirmed using a multi-range pH paper to test the distillate. The pH of the distillate, as indicated by the colour of the pH paper, must be 7. Take care to disconnect the delivery tube from the lower end of the condenser before switching off the heating source. If this is not done, the distillate will be sucked back into the distilling flask, when it starts cooling down, after the removal of the heating source. If, however, arrangements can be made to maintain a flow of nitrogen gas through the distillation assembly, then the back suction problem will not be experienced. Rinse the connecting tube with distilled water and add the rinsings to the receiver beaker. Titrate the contents of the beaker with 0.2 N sodium hydroxide solution using phenolphthalein as indicator. At the end point, colourless solution will turn pink.



All dimensions in millimetres.

FIG. 1 APPARATUS FOR DETERMINATION OF ISOPROTURON CONTENT (ALKALINE HYDROLYSIS METHOD)

A-3.5 Calculations

$$\text{Isoproturon content, percent by mass} = \frac{[(V_1 \times N_1) - (V_2 \times N_2)]}{M} \times 20.6$$

where

V_1 = volume in ml of hydrochloric acid originally taken in the receiver beaker,

N_1 = normality of the hydrochloric acid,

V_2 = volume in ml of sodium hydroxide consumed in the titration,

N_2 = normality of sodium hydroxide, and

M = mass in g of the sample taken for analysis.

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